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VACUUM ULTRAVIOLET MEASUREMENTS OF REFLECTANCE AND LUMINESCENCE FROM VARIOUS ROCK SAMPLES

By

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UNITED STATES
DEPARTMENT OF THE INTERIOR
GEOLOGICAL SURVEY
WASHINGTON, D.C. 20242

Interagency Report
NASA-96
January 1968

Mr. Robert Porter
Acting Program Chief,
Earth Resources Survey
Code SAR - NASA Headquarters
Washington, D.C. 20546

Dear Mr. Porter:

Transmitted herewith is one copy of:

INTERAGENCY REPORT NASA-96

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REFLECTANCE AND LUMINESCENCE FROM

VARIOUS ROCK SAMPLES*

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Sincerely yours,

William A. Fischer
Research Coordinator
Earth Orbiter Program

*Work performed under Work Order No. T-65757
**IIT Research Institute, Chicago, Illinois

UNITED STATES

DEPARTMENT OF THE INTERIOR

GEOLOGICAL SURVEY

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VACUUM ULTRAVIOLET MEASUREMENTS OF REFLECTANCE AND LUMINESCENCE FROM VARIOUS ROCK SAMPLES

I. INTRODUCTION

This study is a continuation of the work reported in "Reflectance of Rocks and Minerals to Visible and Ultraviolet Radiation," Technical Letter, NASA-32, July, 1966, (Ref. 1), in which reflectance data were obtained from 2300A to 7000A with a Cary Model 14 MR recording spectrophotometer utilizing the Model 1411 diffuse reflectance attachment. The overall objective of these studies is to determine if there are significant differences in the spectral reflectance or emission from various geological materials which may be used for identification and/or differentiation purposes in remote sensing applications. The two factors which distinguish the current study are:

1. The samples were in an evacuated chamber during the measurements. This permitted the short wavelength range to be extended into the so-called "vacuum ultraviolet," i.e., wavelengths shorter than 2000A. In order to facilitate comparison with the previous study the spectral region from 2300A to 3000A was repeated.

2. Measurements were made with both monochromatic and broad band sample illumination utilizing a corresponding broad band and monochromatic detection.

This permitted the separation of the reflection and luminescence components, which was not possible in the previous study.

II. EXPERIMENTAL Techniques

A. Sample Surface Preparation

The eleven samples selected for study and their source localities are listed in Table 1. Each sample was cut to form a one inch by one inch slab of about one-quarter inch thickness. One surface of the slab was ground with 320 silicon carbide (30 micron particle size). This surface is designated as the "ground" surface. The other surface was further ground with W8 aluminum oxide (8 micron particle size) and then polished with Linde "B" (0.05 micron) on one-quarter inch thick felt. This surface is designated as the "polished" surface. After grinding or polishing, the surfaces were rinsed in absolute alcohol immediately prior to insertion in the sample measurement chamber. Care was taken so that the sample surfaces were not touched or contaminated in handling.

Except for the pumice sample the surfaces of the eight rocks were the same as those used in the previous study of Reference 1. The pumice could not be polished because of its porous vesicular nature. In the slab size required, the pumice samples were somewhat fragile and, in fact, two samples broke in the process of mounting in the sample holder.

The meteorite samples, especially the Coarse Octahedrite, were more difficult to size and work. In the previous study the meteorite was simply cut into two parts, and the cut surface of one part was ground and the opposite surface part of the curt was polished. The requirement of a small slab sample

Table 1

REFLECTANCE SAMPLES

Code No.	Type	Source Locality
Li 2617	Biotite Granite	Pike's Peak, Colorado
Li 3760	Rhyolite	Nathrop, Colorado
Li 3905	Monzonite	Tintie, Utah
Li 3770	Diorite	Timichi Creek, Gunnison, Colorado
Li 4238	Gabbro	Everton, New York
Li 2288	Basalt	Whatcomb Co., Washington
E 18086	Dunite	Buck Creek, Macon Co., No. Carolina
Li 1832	Obsidian	Regla Falls, Aidalgo, Mexico
Li 1221	Pumice	Latacunga, Equador
Me 1990	Hypersthene Chrondrite (stony)	Colby, Clark Co., Wisconsin
Me 1252	Coarse Octahedrite (iron-nickel)	Canon Diablo, Coconino Co., Arizona

to fit into the vacuum sample chamber in this work required more cutting and in so doing the actual surfaces used were not the same as those previously examined. The Coarse Octahedrite (iron-nickel) appeared as a metallic surface which took a high polish; the Hypersthene Chondrite surface had a mottled appearance with some small, shiny metallic spots.

B. Instrumentation

A Jarrell Ash Model 78-650 one-half meter scanning vacuum grating monochromator of the Seya-Namioka type was used. The grating size is 38 x 38 mm with 30,000 lines per inch, blazed for use in the 500A to 3000A range. Monochromator slit widths of about 0.3 mm were used, which with the instrument dispersion of 17A per mm gave a band pass of about 5A.

The source was a Tanaka-Type capillary discharge tube (Jarrell Ash Model 45-202) exciting ultra pure (99.999%) hydrogen at 15,000 volts AC and 500 milliamps. A windowless configuration was used, i.e., the source gas was differentially pumped through the entrance slit of the monochromator. In normal operation the monochromator chamber pressure was 10^{-3} mm Hg and the source chamber was filled with hydrogen at about 0.5 to 1 mm pressure.

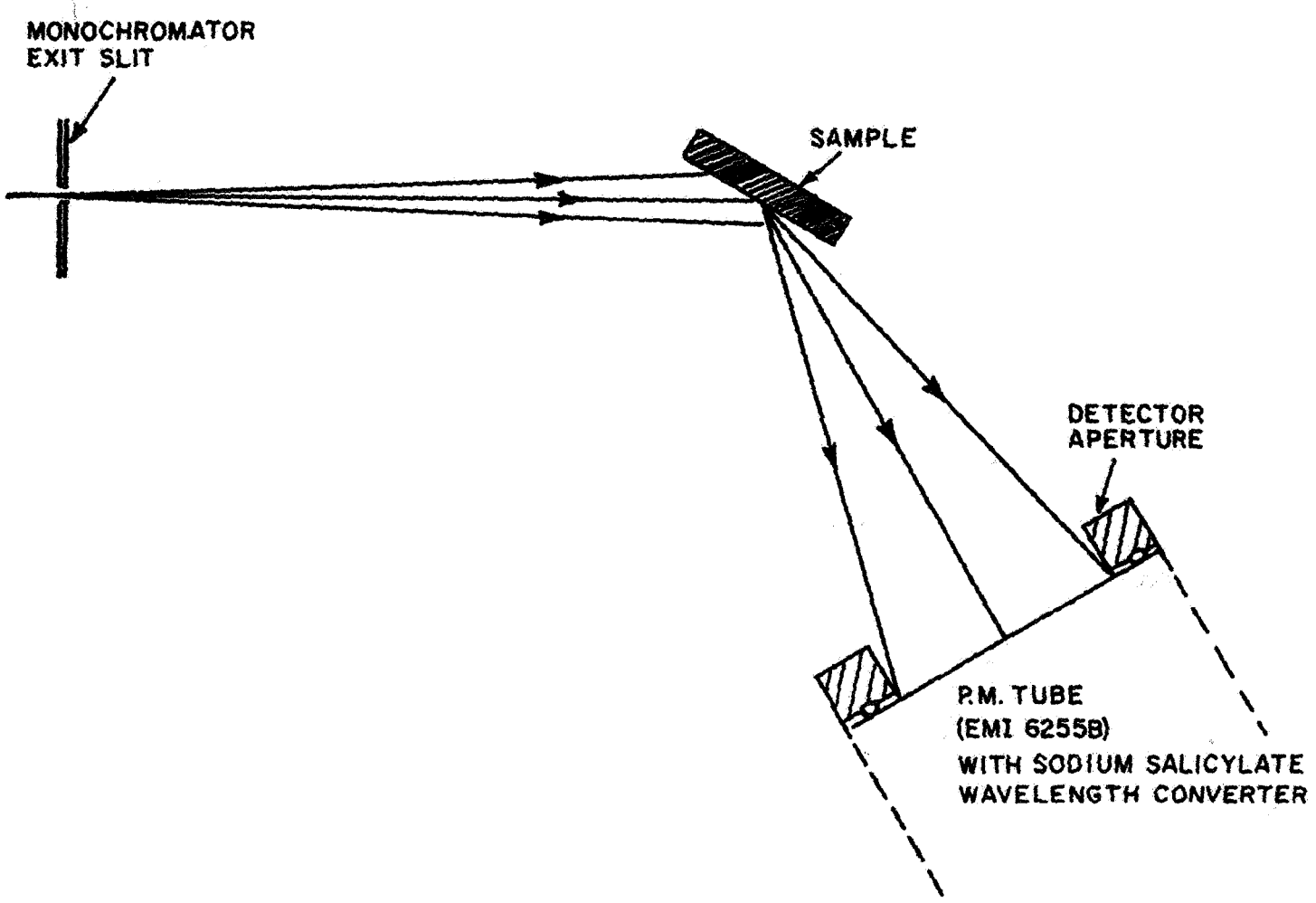
The detector was an EMI 6255B multiplier phototube with an S-13 response. A thin coating of sodium salicylate was used to extend the standard S-13 response in visible and near ultraviolet to wavelengths as short as 900Å in the vacuum ultraviolet.

The associated circuitry, high voltage supply, micro-micro ammeter and recorder, were the standard Jarrell Ash instrument package. The multiplier phototube had a dark current of the order of 10^{-11} amps at 1000 volts. Most of the data measurements produced signals of at least 10^{-9} amps so that the detectability was generally good.

C. Measurement Geometry

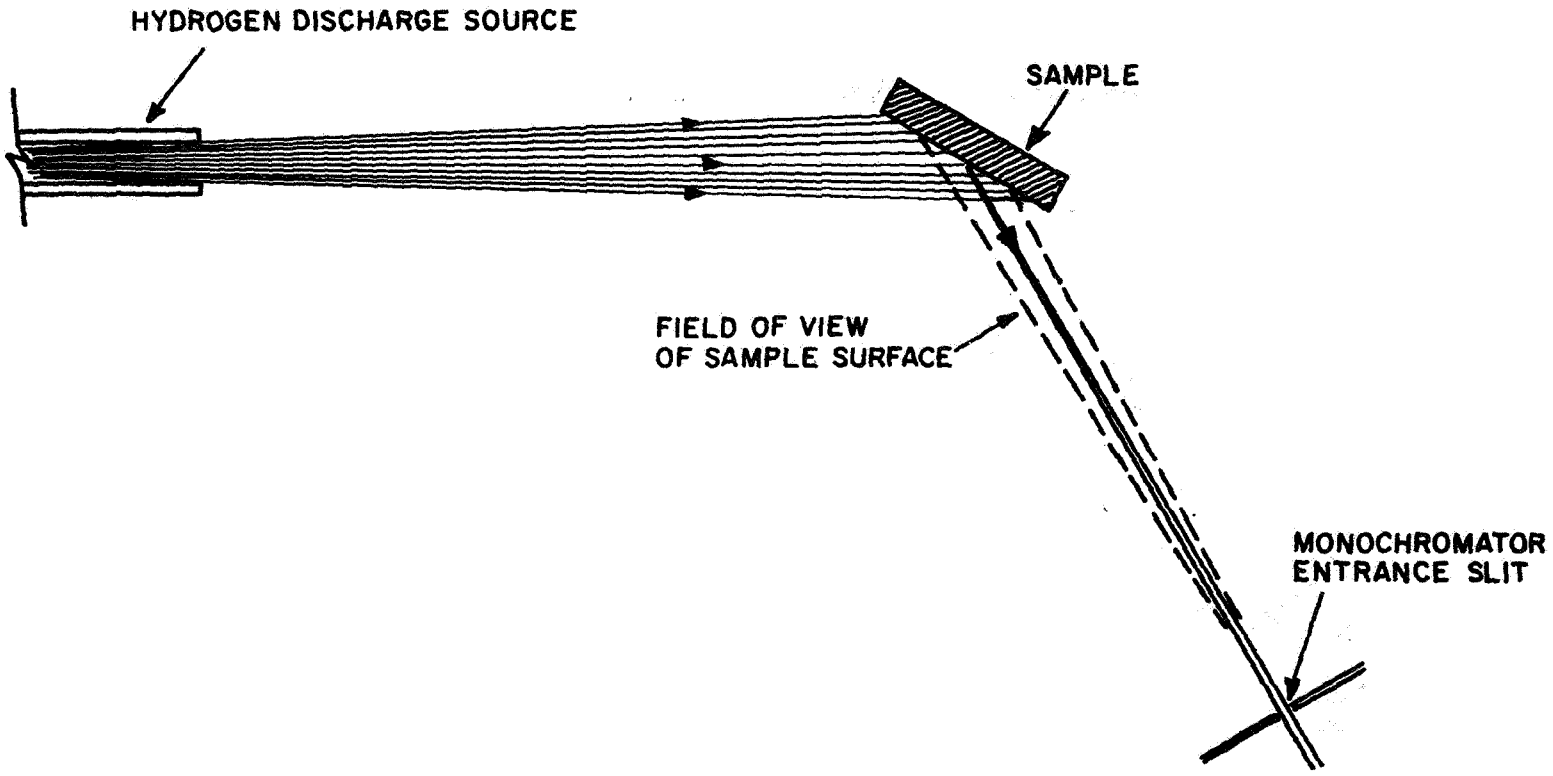
A vacuum tight sample chamber was constructed to hold six samples on a rotatable hexagonal wheel. This chamber could be attached to either the entrance or exit slit of the monochromator; either the source or the detector could be attached to the other face of the sample chamber. The hexagonal wheel permitted each sample to be rotated into measurement position. The remaining five samples in the chamber were completely masked off from a direct view of either the source or detector. A black matte liner on all inside surfaces of the chamber served to reduce light scattering, and a light baffle was built in to block the direct line of sight between source and detector.

The two types of measurement geometries are illustrated in Figures 1 and 2. In both geometries the illumination and detection angles are 60 degrees to the sample surface normal. In the Type I geometry (Figure 1) a 10 x 15 mm sample surface area is illuminated with focused, monochromatic light. The sample surface is viewed by the detector though a circular aperture of 3.6 cm diameter at a centerline distance of



GEOMETRY FOR TYPE I MEASUREMENTS

FIGURE 1.



GEOMETRY FOR TYPE II MEASUREMENTS

FIGURE 2.

7.4 cm between sample and detector. Because of the broad spectral sensitivity of the detector, the recorded signal in this case includes both the sample reflectance at the incident monochromatic wavelength and any ultraviolet or visible (up to about 6000A) luminescence which the incident wavelength excites.

In the type II geometry (Figure 2) the entire sample surface is illuminated with the full hydrogen source spectrum. The sample reflectance plus luminescence is then detected with spectral selectivity through the monochromator. The monochromator field of view covers a sample surface area of about 14 mm x 10 mm. Each element within this surface area, however, is detected with a very small acceptance angle defined by the entrance slit of the monochromator (0.3 mm x 10 mm) at a center line distance of 8.4 cm between sample and slit.

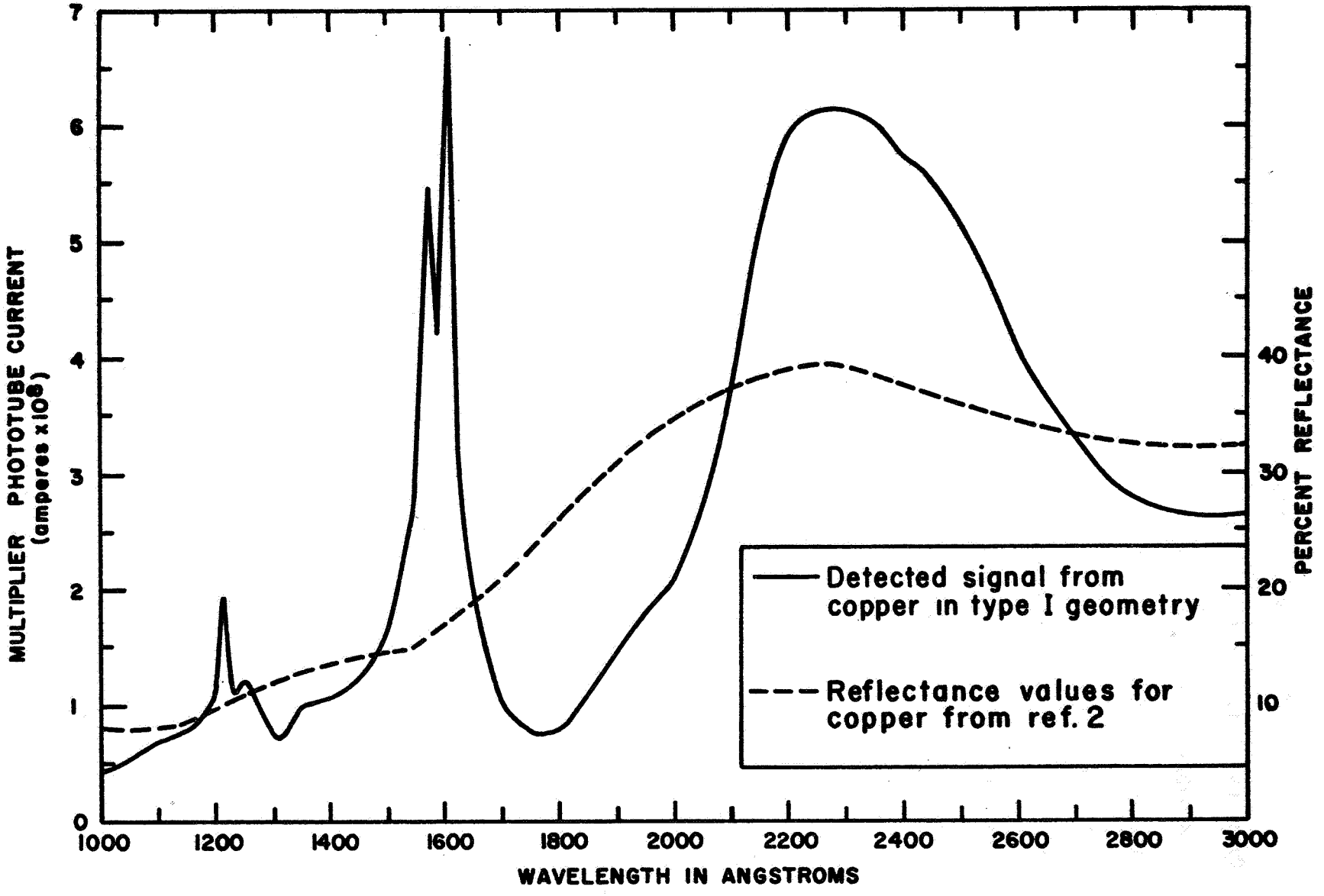
D. Measurement Methods and Standardization

The sample chamber was loaded with either five or four samples and one or two reference materials for each set of measurements. The entire unit, monochromator and sample chamber, was pumped down to 10^{-5} mm or less pressure and outgassed for at least two hours before the hydrogen gas flow to the source was begun. When a stable hydrogen gas flow had been reached (with the hydrogen flow the monochromator pressure is about 10^{-3} mm), the source is ignited and allowed to stabilize. In the type II

geometry, where the sample chamber is between the source and the monochromator, some differential pumping is used between the source and samples to reduce the hydrogen content at the samples.

Most of the data were obtained by recording the detector signal as the monochromator was scanned from 3000A down to 1000A at a scan rate of 100A per minute. In all cases a reference piece of polished aluminum was in the sample chamber and was scanned intermittently between sample scans. The signal data for the sample scans were then normalized to the aluminum reference signal at the corresponding wavelengths. The function of the aluminum reference was primarily to monitor the source output. Absolute reflectance values were eventually derived on the basis of a polished copper piece which was compared to the aluminum reference. The values of Ehrenreich and Phillipp (Ref. 2) for the absolute reflectance of polished bulk copper in the vacuum ultraviolet were used to calibrate the aluminum reference.

The detected signal (multiplier phototube current) versus wavelength from the polished copper reference in Type I geometry is shown in Figure 3. Also plotted in this figure is the copper spectral reflectance curve which was used as the standard. The detected signal curve shape is a function of the hydrogen source spectrum, the detector spectral response, and the sample reflectance. Two features of the hydrogen source are prominent on all of the raw data curves; these are the hydrogen Lyman line at 1215A and the high intensity peaks around 1600A.



COPPER REFERENCE DATA

FIGURE 3.

The final standardization of the reflectance values was confused at first when a comparison of the aluminum reference and the copper standard gave relative values which were not compatible with their literature values. Further experiments utilizing fresh aluminum and copper samples were then performed to establish the correct reflectance value standard. These experiments were successful in explaining the early discrepancy as being due to a degradation of the aluminum reference sample reflectance caused by the hard ultraviolet radiation. Fortunately, the original aluminum reference had been degraded during the very first checkout runs prior to the sample data runs so that the aluminum remained as a constant secondary reference to which the copper standard could be correlated.

To verify the reproducibility of test results, data on three samples were taken twice by scanning runs on separate days and once at fixed wavelength points, where the wavelength was held constant while the detected signals were measured alternately from the samples and from reference materials in the chamber. The final curves derived from these samples had essentially the same shapes for each of the three measurements.

III. DATA AND DISCUSSION

In Table 1 the eight rocks are listed in order of the generally expected decreasing silica content, from the acidic biotite granite and rhyolite to the ultra-basic dunite. Detailed compositional and surface texture analyses have not been performed; however, these samples are available if such studies are warranted.

The normalized data curves obtained for both Type I and Type II geometries of the same sample surface are presented in the Appendix. Data points were analyzed and plotted at 50 Angstrom intervals except for wavelengths near 1200 A and 1600 A where the source input maxima produced such sharp slopes in signal change on the recorded scans (see, for example, Figure 3), that it was not possible to obtain a precise data point.

To determine the fraction of energy collected at the detector from the total emission reflection by the rock surface, the detector solid angle was computed and compared

to the total angle of emission or reflection. The estimated fractions from Figures 1 and 2 are specified below:

a) Type I, diffuse reflectance -- 0.06

b) Type I, luminescence emission -- 0.03

c) Type II, diffuse reflectance -- 2×10^{-4}

d) Type II, liminescence emission -- 1×10^{-4}

In both geometries the sample surface normal was adjusted and fixed so that the source and detector angles to the normal satisfied the law of specular reflection; this was observed by a peaking of the response of the detector. Thus the detection fractions for the specularly reflected components are assumed to be unity for both geometries.

Using the above detection fractions we can write the equations for the relative components of the detected signals. No specular reflectance component is included in the "ground" surface measurements equations.* The signal equations are written below with the designations (P-I), (P-II), (G-I), and (G-II) wherein the symbols P and G refer to the polished and ground surfaces, and the symbols I and II refer to the measurement geometries.

$$(P\text{-}I)_\lambda = R_s(\lambda) + 0.06\ R_d(\lambda) + 0.03\ L_I \qquad (1)$$

$$(P\text{-}II)_\lambda = R_s(\lambda) + 2 \times 10^{-4}\ R_d(\lambda) + 10^{-4}\ L_{II} \qquad (2)$$

---

* The data of Reference 1 showed no significant differences in ground surface reflectance curves with or without the inclusion of the specular reflectance component.

$$(G\text{-}I)_\lambda = 0.06\ R_d^1(\lambda) + 0.03\ L_I \quad (3)$$

$$(G\text{-}II)_\lambda = 2 \times 10^{-4}\ R_d^1(\lambda) + 10^{-4}\ L_{II} \quad (4)$$

where:

$R_s(\lambda)$ = specular reflectance at wavelength λ,

$R_d(\lambda)$ = diffuse reflectance at wavelength λ, from the polished surface,

$R_d^1(\lambda)$ = diffuse reflectance at wavelength λ, from the ground surface,

L_I = luminescence excited by narrow band (5A) energy increment at λ and detected with broad band detector, and

L_{II} = luminescence excited by direct broad band source and detected with narrow band (5A) resolution at λ.

The L_I signal component as a function of wavelength is the luminescence excitation spectrum. In general for mineral or inorganic luminescent materials, the excitation spectra will have a threshold and then increase as the wavelength decreases to a nearly constant efficiency over a broad range of wavelengths.

The L_{II} signal component as a function of wavelength is the luminescence emission spectrum. Luminescence emission will occur at longer wavelengths than the excitation wavelength. In a simple luminescent system the emission spectrum is a band with a width at half maximum intensity of several hundred Angstroms. Materials such as rocks may

have several overlapping emission bands and a very broad and/or complex spectrum.

Examination of the components contributing to the normalized measured quantities (left-hand sides of Equations 1-4), leads to the following:

1. The extremely small coefficients modifying the diffuse (R_d) and luminescence (L_{II})contributions to the (P-II) measurements, Equation 2, tend to remove these contributions in this measurement. Therefore, it is highly likely that the (P-II) curves shown in the Appendix exhibit predominantly the specular reflectance properties of the polished rock samples.

2. The (G-II) data curves are the most favorable sources of information for detecting luminescence. The ground-surface measurements, Equations 3 and 4, remove a major contribution from specular reflection, in contrast to those from the polished surface, Equations 1 and 2. Thus, the ground-surface measurements leave only the diffuse reflection component to compete with the luminescence emission in data interpretation. A comparison of the L_I and L_{II} intensities can be deduced directly from Equations 3 and 4; the effects of differing source intensities employed in the two geometries have been removed in these equations by normalization of the (G-I) and (G-II) values measured.

Solving Equations 3 and 4 simultaneously to remove the diffuse reflectance contributor, R_d^1, one obtains,

$$L_{II} - L_I = 10^4(G\text{-}II)_\lambda - 33(G\text{-}I)_\lambda \qquad (5)$$

For all rock samples investigated, the normalized data values

indicate that,

$$(G\text{-}I)_{\lambda} \leq 43(G\text{-}II)_{\lambda} \tag{6}$$

So that, in all cases L_{II} is greater than L_I and therefore,

$$\frac{L_{II}}{R_d^I} > \frac{L_I}{R_d^I} \tag{7}$$

which indicates the advantage of the (G-II) data curves over the (G-I) data for detection of luminescence. The Appendix curves for the ground surfaces tend to corroborate this since the Type II geometry data exhibits considerably more peaking than does the Type I data. Such peaking has been interpreted as probable evidence of luminescence.

Polished Surface - Type I Geometry: 1000-1800 A, a large continuous increase in value with inflections around 1400 and 1600; 1800-2000 A, a broad maximum; 2000-3000 A, a moderate continuous decrease in value.

Ground Surface - Type I Geometry: 1000-2000 A; similar to Polished - Type II; 2000-3000 A values increase with gradual slope (essentially an inversion of Polished - Type II behavior).

Ground Surface - Type II Geometry: Note that these signals are relatively low and subject to larger errors in data points. There is a resolved maximum peak at 2400 A in all samples and a secondary maximum around 1800 A. An inflection or peaking at 2050 is also indicated. Any structure in curve shape below 1700 is questionable.

2. Group 2 - Monzonite, diorite, obsidian

Polished Surface - Type II Geometry: 1000-2050 A, continuously increasing values rising to a sharp peak at 2050; 2050-3000, same as Group I.

Polished Surface - Type I Geometry: 1000-1800 A, a very pronounced maximum peak about 1650; 1800-3000 A, same as Group 1.

Ground Surface - Type I Geometry: generally similar to Group 1 except for obsidian whose value stays about constant from 1800-3000 A.

Ground Surface - Type II Geometry: monzonite and diorite have their maximum peak at 2050 with an indication of

a secondary peak around 2400. Obsidian's curve shape is similar to Group 1, in contrast to its polished surface behavior.

3. Rhyolite and Gabbro

Polished Surface - Type II Geometry: rhyolite tends toward Group 2 shape while gabbro tends toward Group 1 shape.

Polished Surface - Type I Geometry: Both samples look most similar to Group 2 curves.

Ground Surface - Type I Geometry: similar to Group 1 shape rhyolite has a sharp break at 2400 with a very strong increase up to 3000.

Ground Surface - Type II Geometry: same as Group 1 curves.

4. Pumice

Type I Geometry: generally similar curves to Group 1.

Type II Geometry: the pumice surface number 3 (see curve (1, 3) fits the Group 1 curve shape; pumice surface number 2, however, has an anomalous shape intermediate to the two basic groups.

5. Meteorites - Hypersthene Chondrite and Coarse Octahedrite

As mentioned earlier these samples had surfaces different from the rocks. Also the measurements were only performed in the point by point technique at 100 A intervals.

Hypersthene Chondrite (mottled surface): Type I geometry between 1000-2600 A appears similar to Group 1 - ground

surface data with a sharp increase around 1800; 2600-3000, values decrease similar to polished surface rock data. Type II geometry curve is unique with a broad maximum peaking at 2300 A.

Coarse Octahedrite (high metal-like polish): Type I geometry curve appears like a polished metal reflectance curve, note the high reflectance values. Type II geometry curve does indicate some possible structure, more data points would be necessary to confirm this.

IV. OBSERVATIONS

1. All samples, both ground and polished surfaces, have increasing reflectance values as the wavelength increases between 1000 A to 2000 A. The predominant increase occurs between 1600 A and 2000 A.

2. As the wavelength increases from 2000 A to 3000 A, the spectral reflectance decreases for polished surfaces and increases for ground surfaces. The largest relative differences in slope values of the curves occur for the ground surfaces.

3. The gross aspects of the ground surfaces are similar in that one or more relatively sharp peaks and changes in slope direction are common for all ground surfaces. Fewer sharp peaks and changes in slope are noted for the polished surfaces. These and other similarities in gross aspects of the curves suggest that surface texture rather than composition are major factors in determining spectral reflectance, particularly at wavelengths shorter than 2000 A.

4. The general curve characteristics for monzonite and diorite are markedly similar to each other, and likewise, markedly dissimilar to rocks of more acidic and basic composition. However, curve characteristics of other rock types bear no relation to composition. For example, curves for polished biotite granite are more similar to curves for dunite, an ultrabasic rock unrelated compositionally, than they are to rhyolite which is compositionally similar to granite.

5. All rocks showed a luminescence emission at about 2400 A, while monzonite and diorite had their peak emission at 2050 A. The high source intensity around 1600 A was distinctly exciting luminescence, but without a correlation with the emission bands it is not possible to estimate the observable emission under solar illumination.

V. CONCLUSIONS AND PLANS FOR FUTURE WORK

Although initial analyses of monzonite and diorite at wavelengths shorter than 3000 A suggest a correlation between composition and UV spectral response, other rock samples of more acidic and basic composition displayed little or no correspondence with their spectral reflectance curves. Quite possibly this lack of correspondence is due to differences in surface texture between the sample surfaces. Although identical abrasives were used to prepare all sample surfaces, it is possible that the standard rock polishing methods used are inadequate, especially at wavelengths shorter than 2000 A where irregularities in the surface may be comparatively large with respect to wavelength.

It is also possible that surface preparation of the samples may somehow modify the molecular structure of the surface layer so that the emission spectrum is altered or quenched. It would be desirable in future work to include several samples ranging from highly polished to finely powdered ($\sim$50 A), prepared from the same specimen, in order that the effect of surface texture on both reflectance and emission may be studied.

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2. H. Ehrenreich and H. R. Phillipp, "Optical Properties of Ag and Cu", Phys. Rev. 128, No. 4, 1622 (1962).

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APPENDIX

DATA CURVES

APPENDIX

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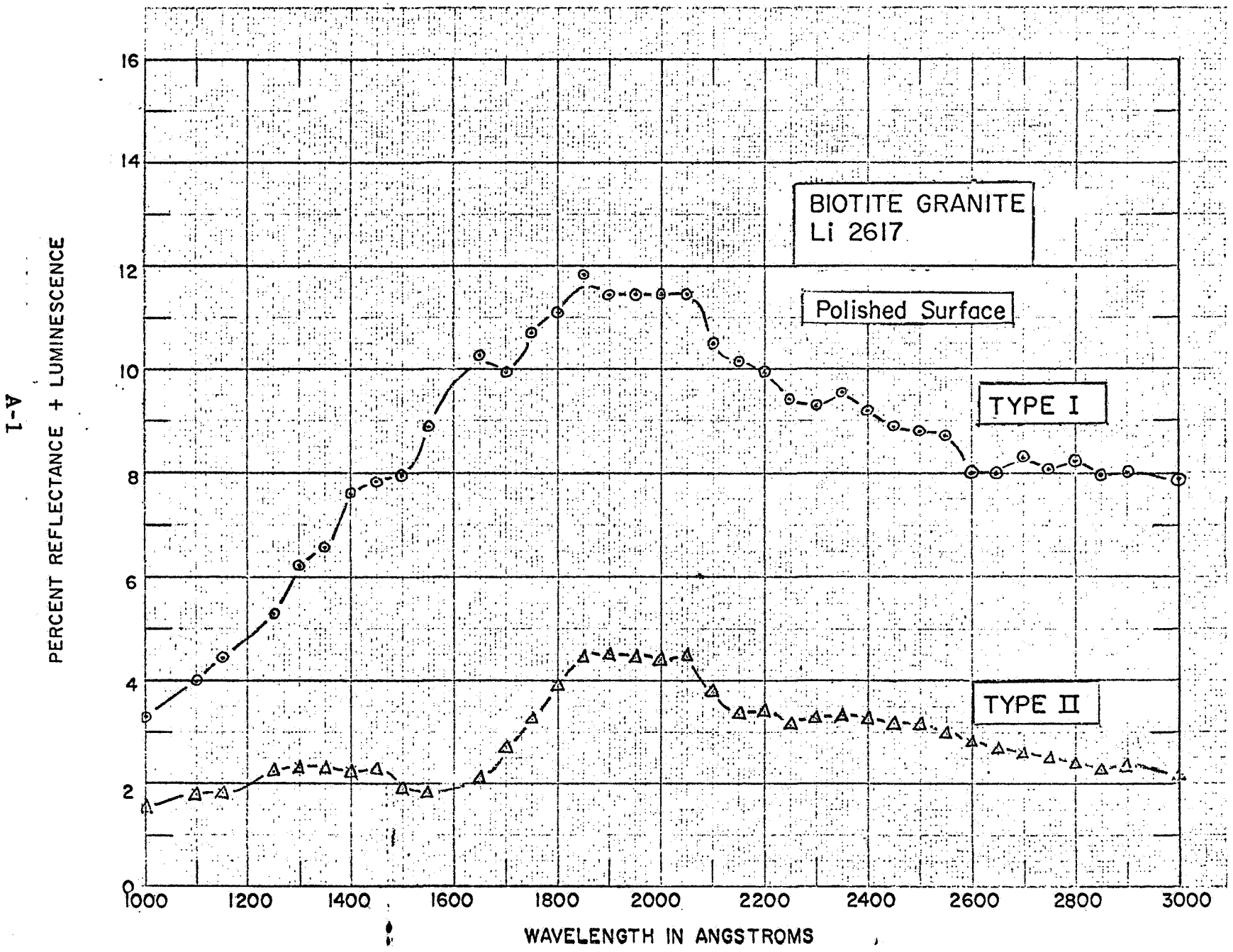
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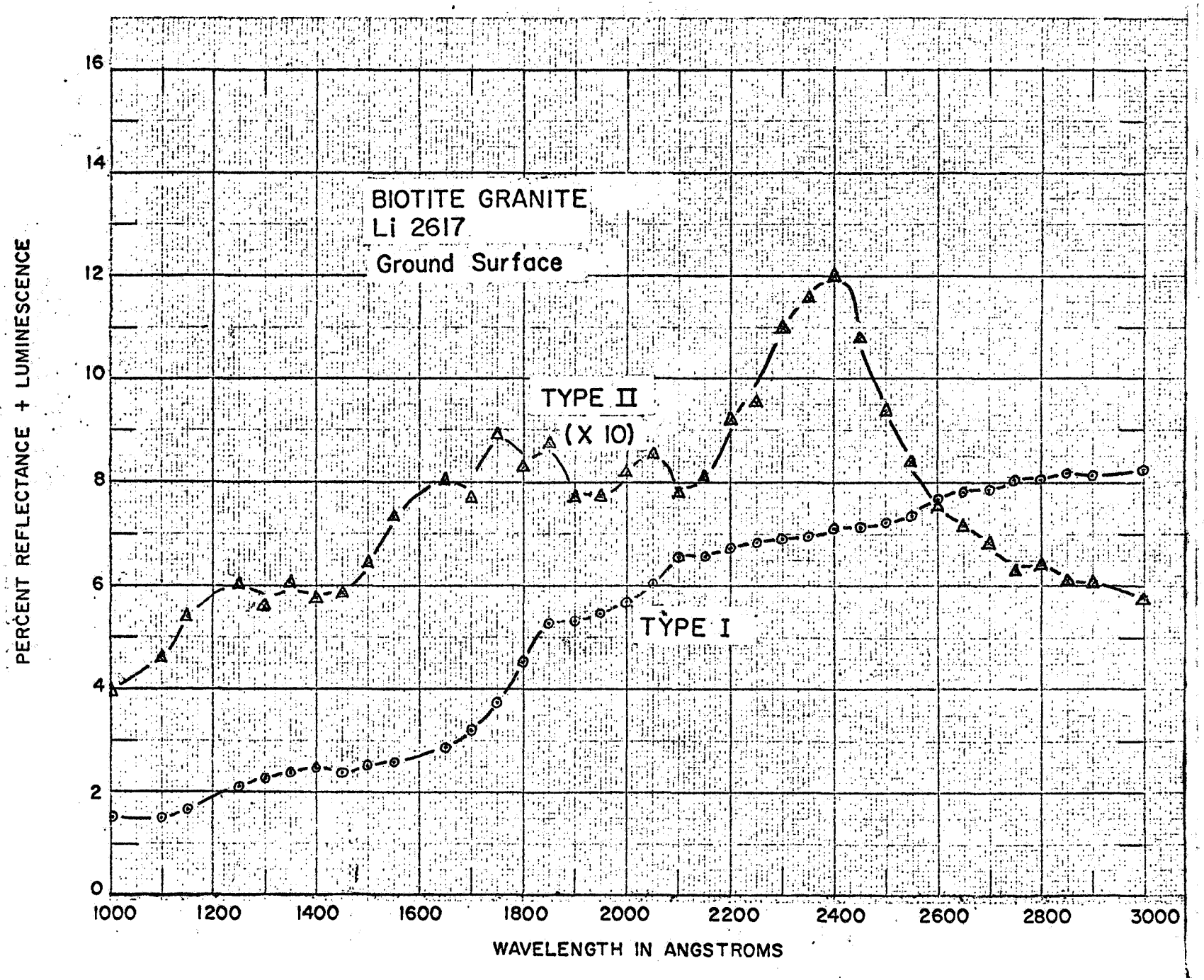
3000

A-1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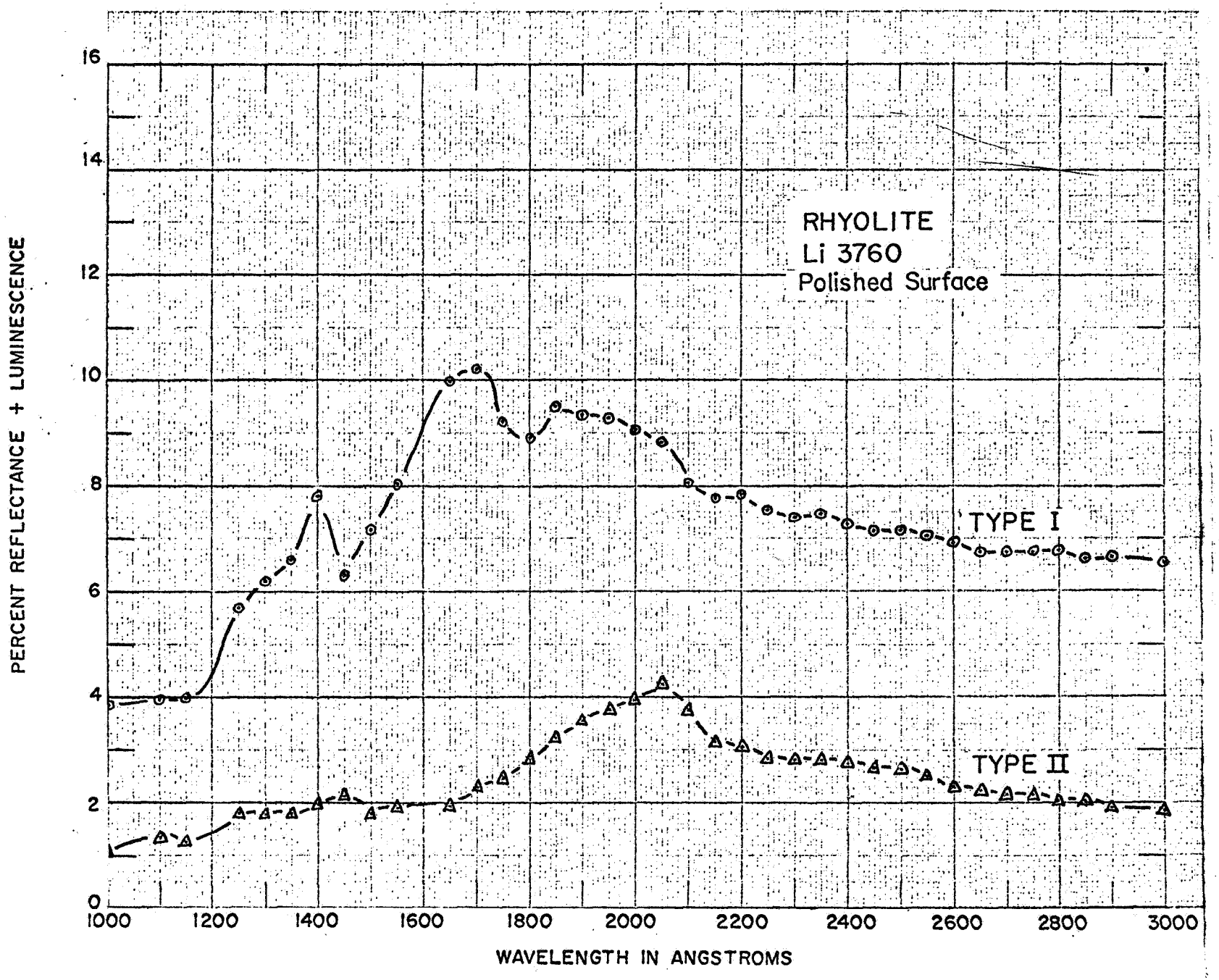
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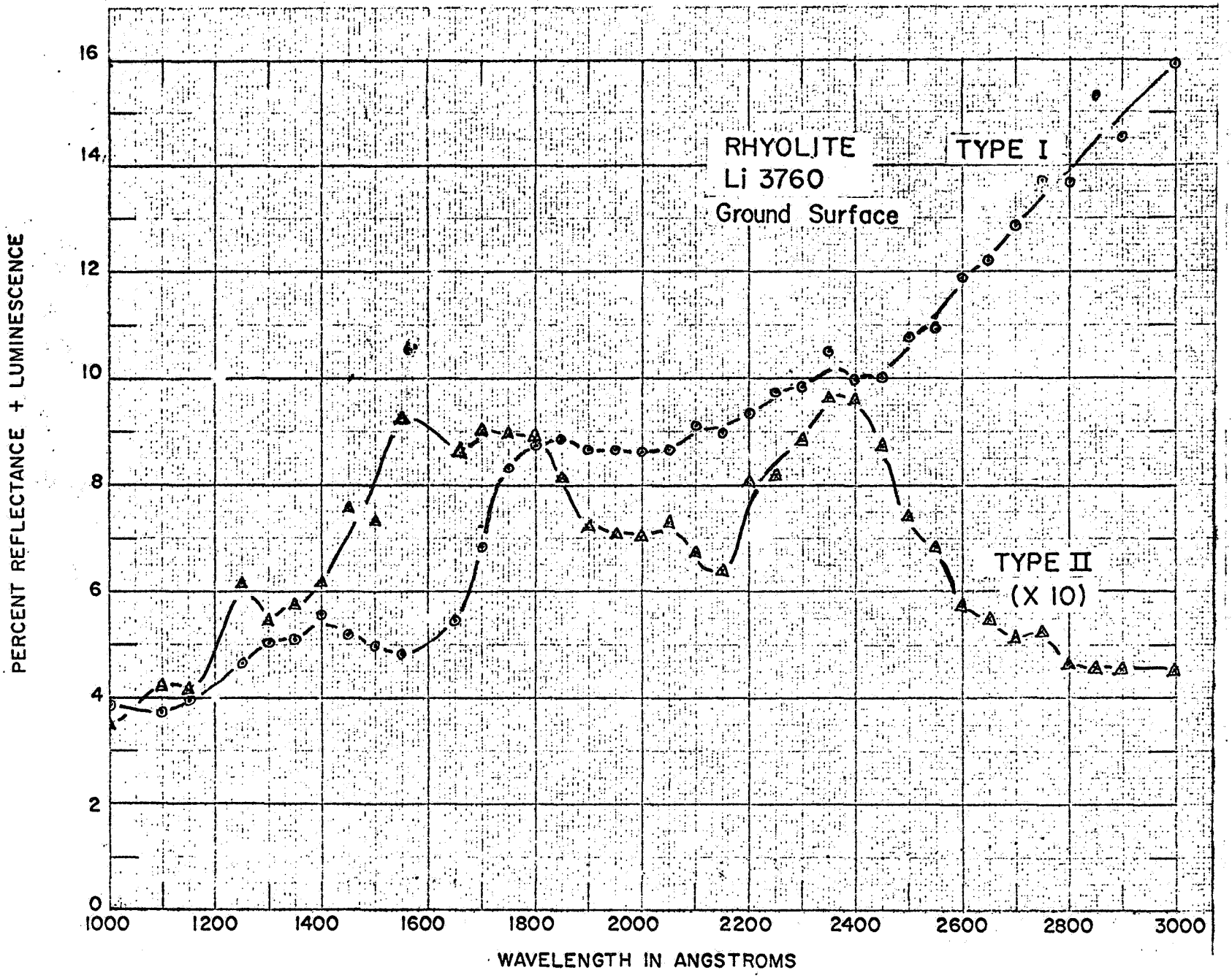
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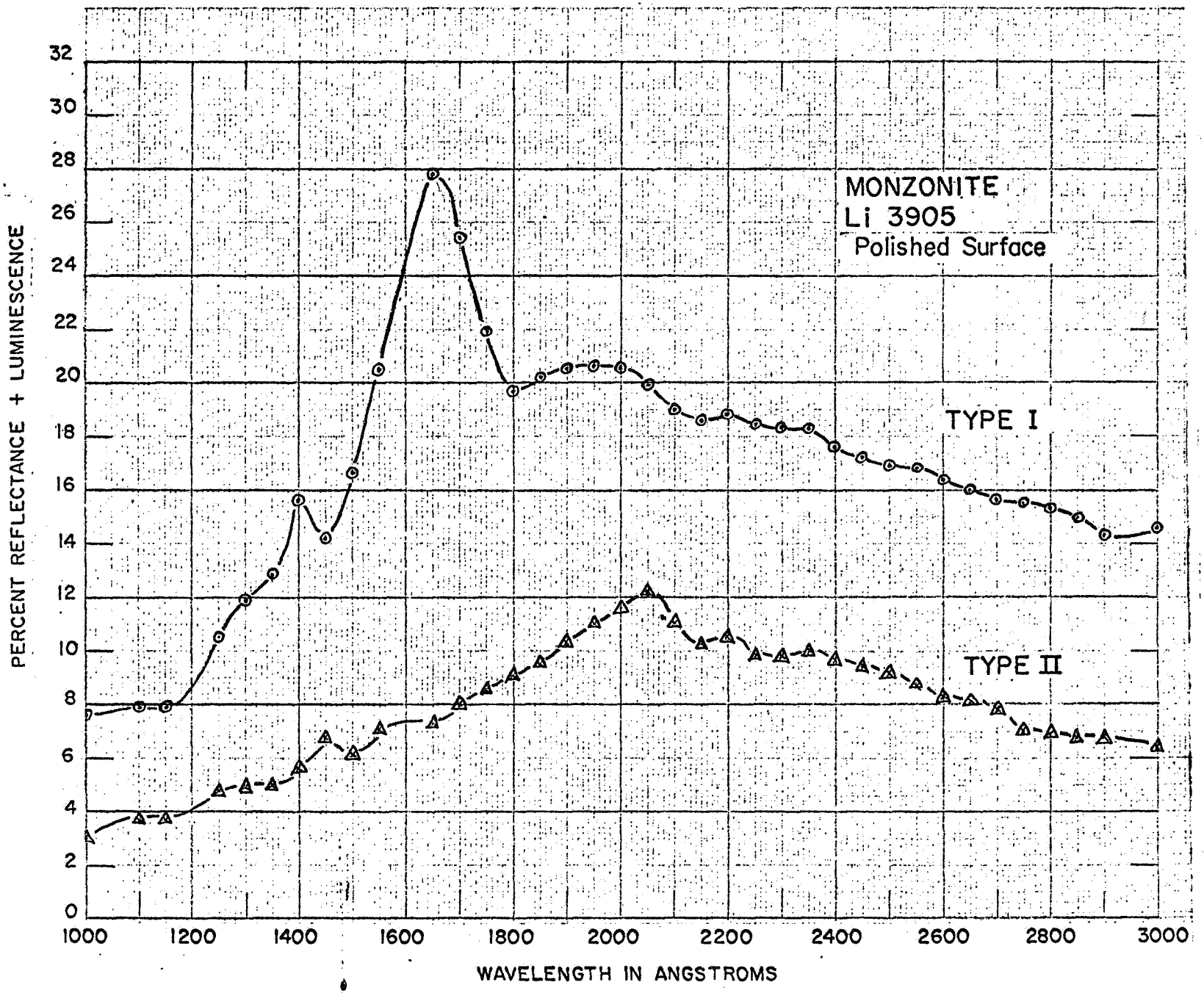
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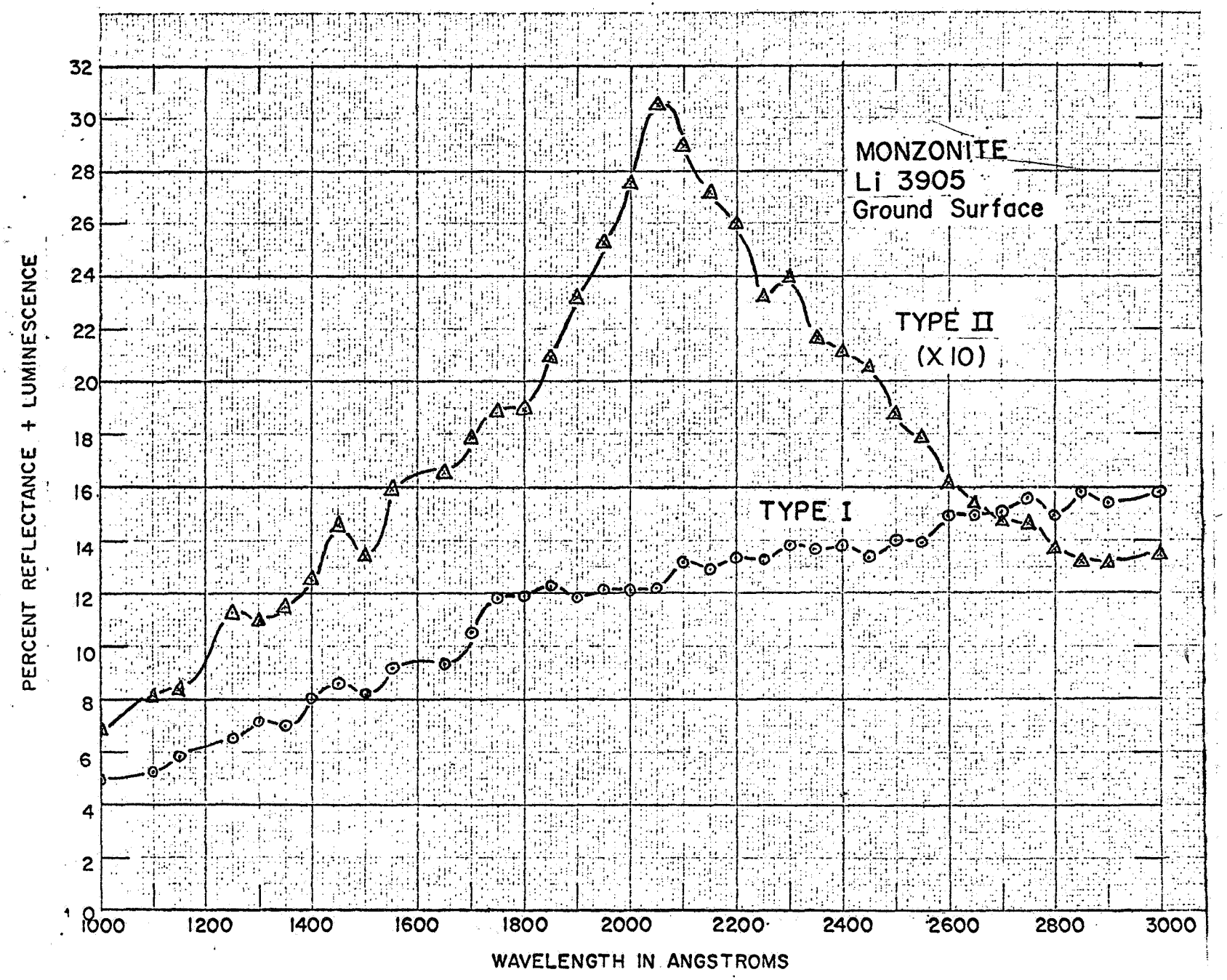
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Li 3905
Ground Surface
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(X 10)
TYPE I
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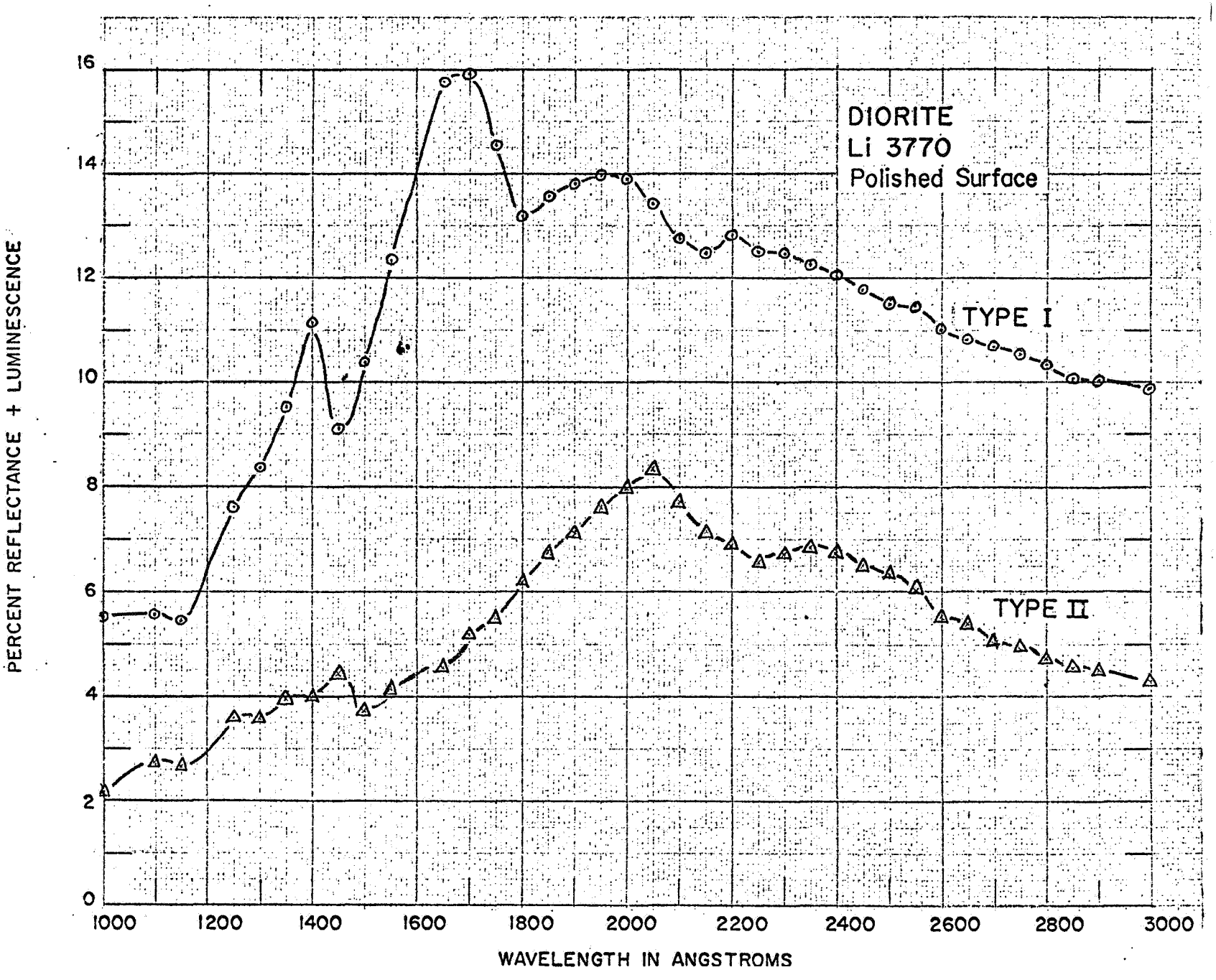
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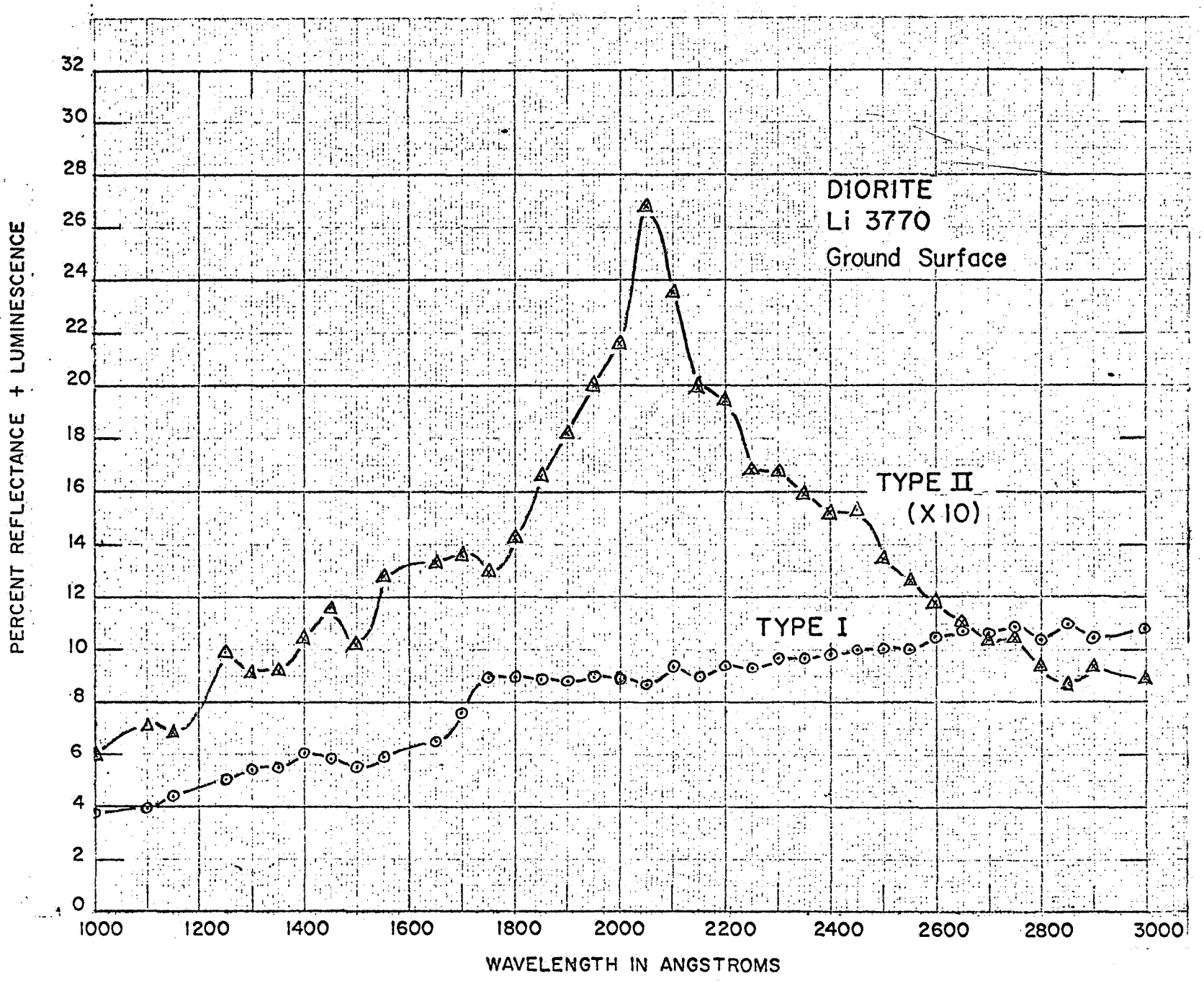
DIORITE
Li 3770
Ground Surface
TYPE II
(X 10)
TYPE I
PERCENT REFLECTANCE + LUMINESCENCE
WAVELENGTH IN ANGSTROMS
0
2
4
6
8
10
12
14
16
18
20
22
24
26
28
30
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1000
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1400
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1800
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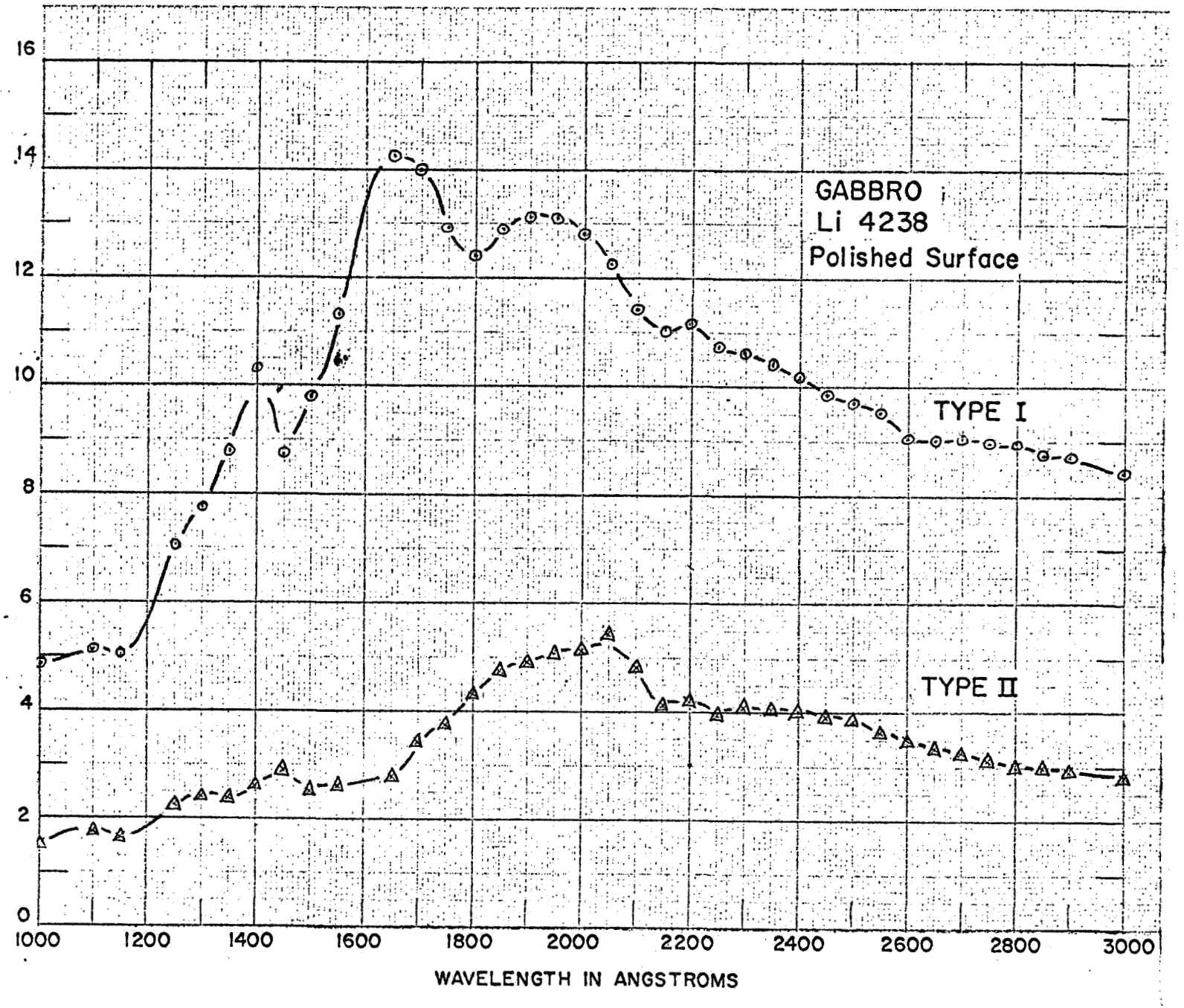
2400
2600
2800
3000


GABBRO
Li 4238
Polished Surface
TYPE I
TYPE II
PERCENT REFLECTANCE + LUMINESCENCE
WAVELENGTH IN ANGSTROMS
0
2
4
6
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10
12
14
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1000
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1800
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2200
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2600
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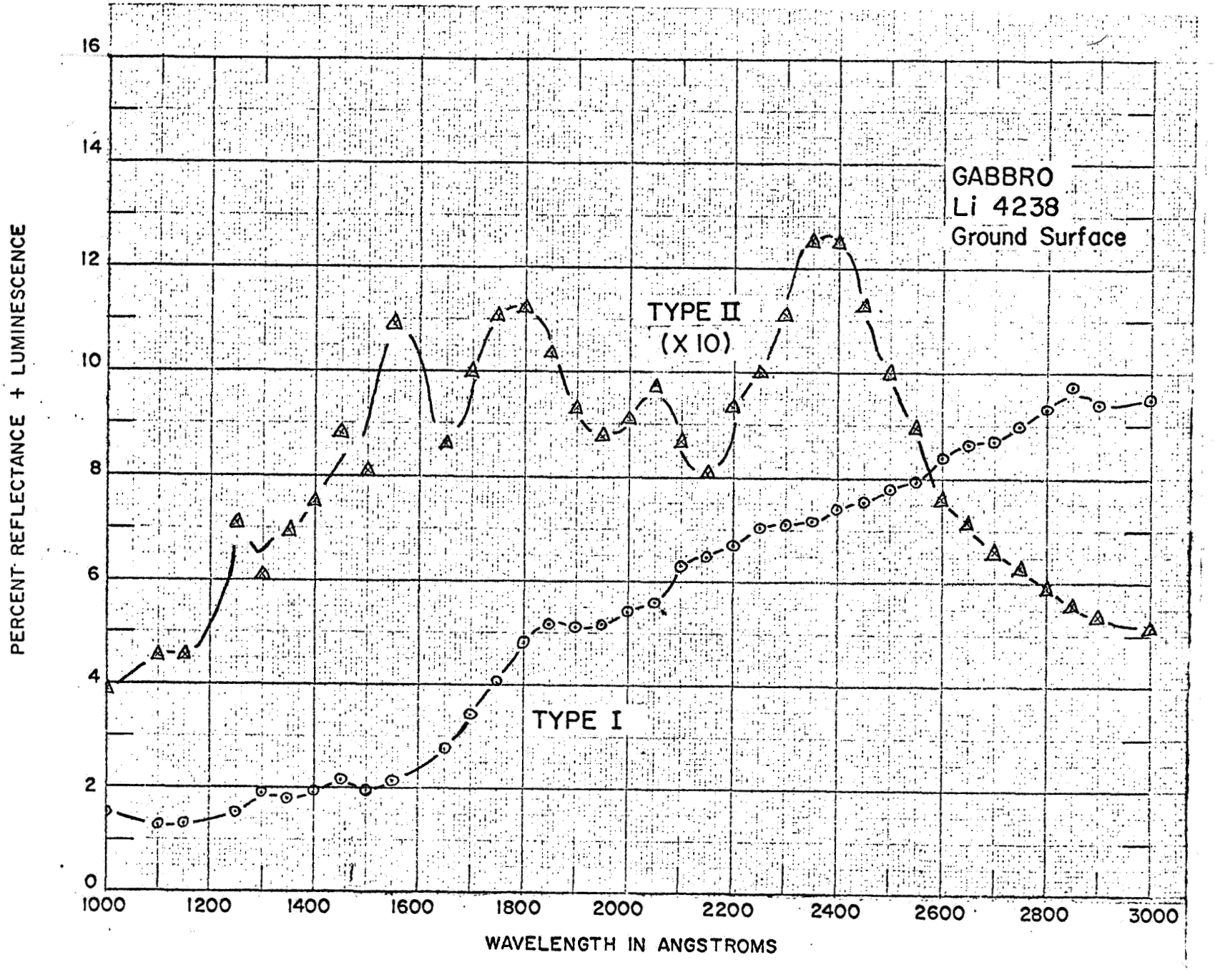
GABBRO
Li 4238
Ground Surface
TYPE II
(X 10)
TYPE I
PERCENT REFLECTANCE + LUMINESCENCE
WAVELENGTH IN ANGSTROMS
0
2
4
6
8
10
12
14
16
1000
1200
1400
1600
1800
2000
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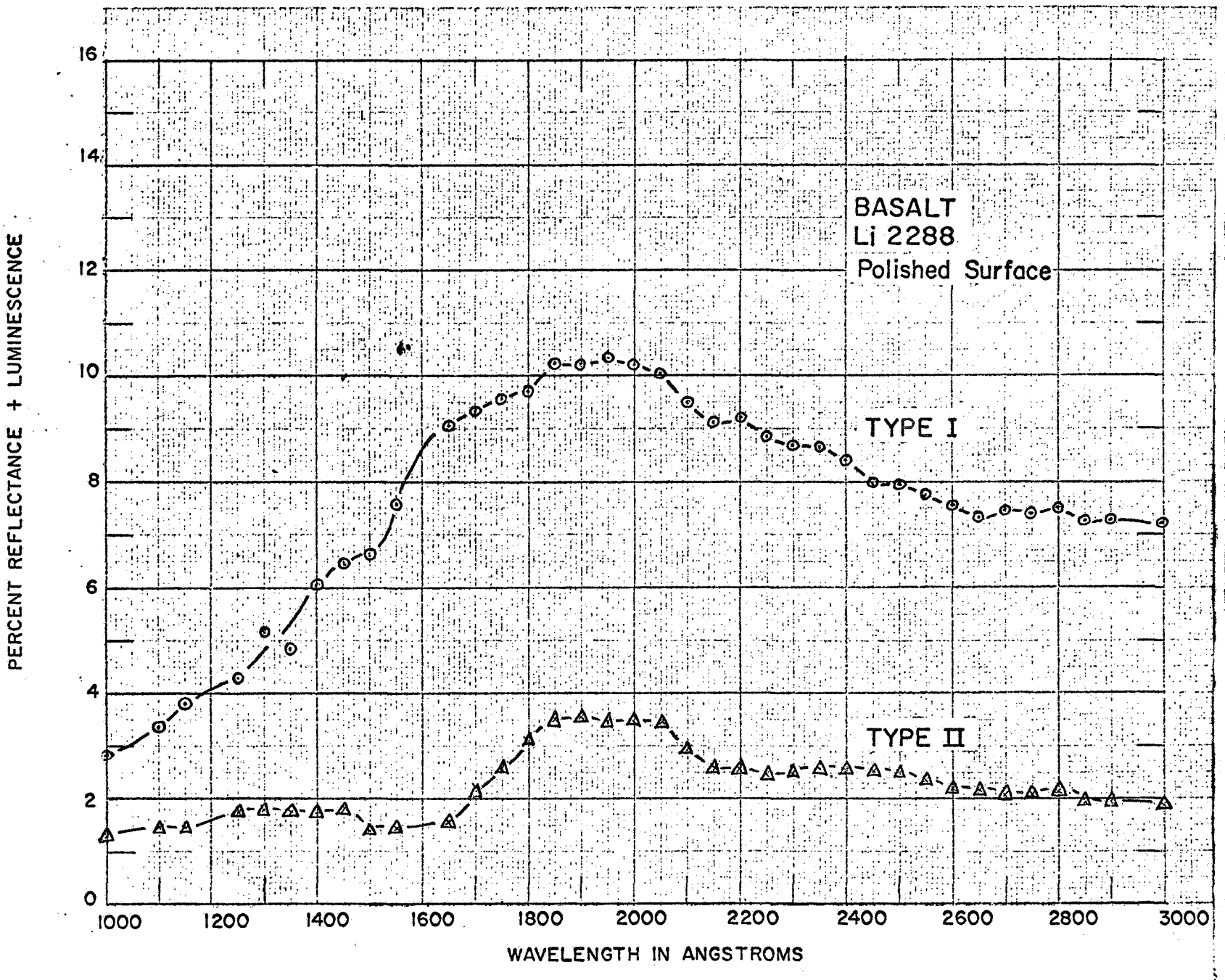
2400
2600
2800
3000


BASALT
Li 2288
Polished Surface
TYPE I
TYPE II
PERCENT REFLECTANCE + LUMINESCENCE
WAVELENGTH IN ANGSTROMS
0
2
4
6
8
10
12
14
16
1000
1200
1400
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1800
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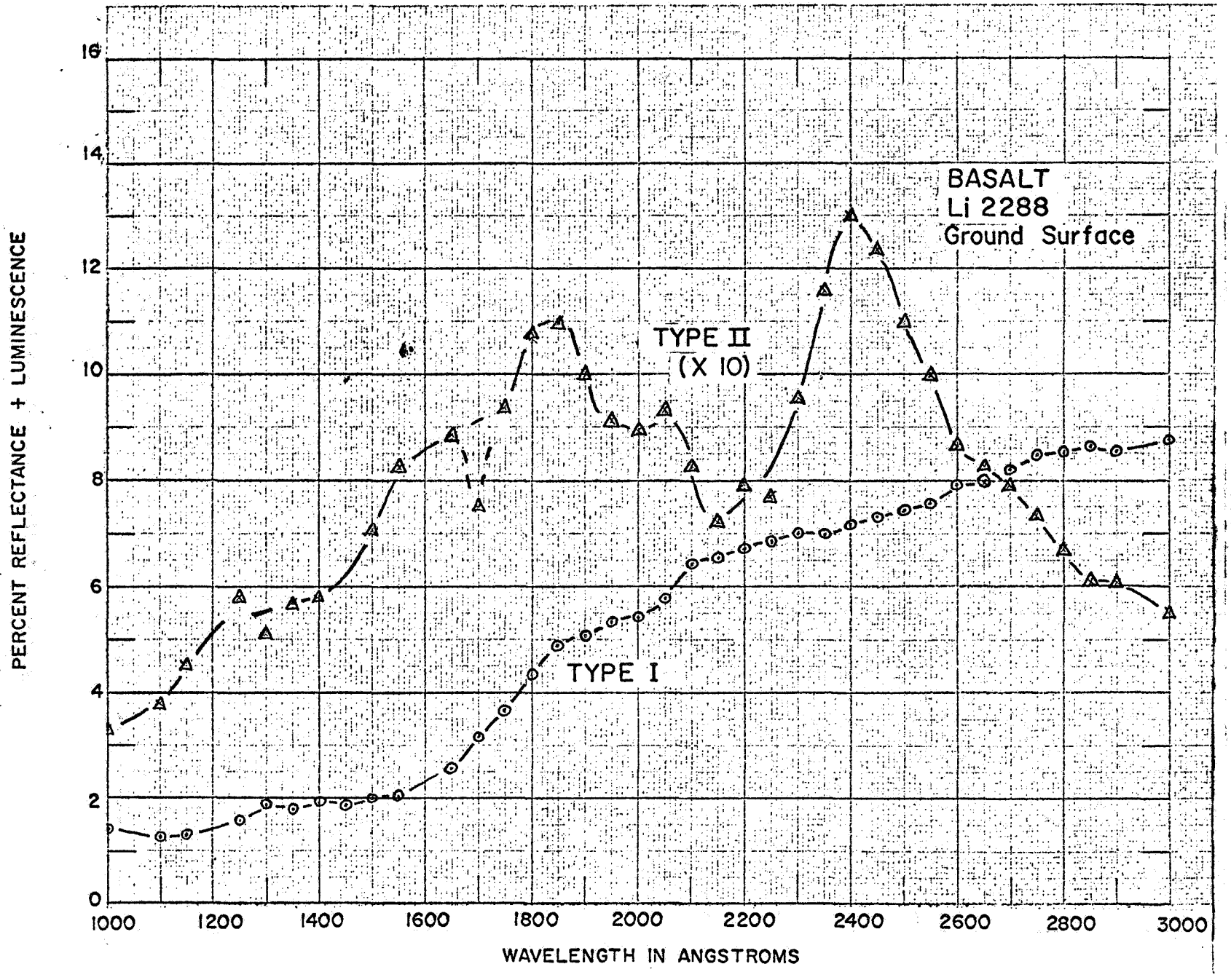
2200
2400
2600
2800
3000


BASALT
Li 2288
Ground Surface
TYPE II
(X 10)
TYPE I
PERCENT REFLECTANCE + LUMINESCENCE
WAVELENGTH IN ANGSTROMS
0
2
4
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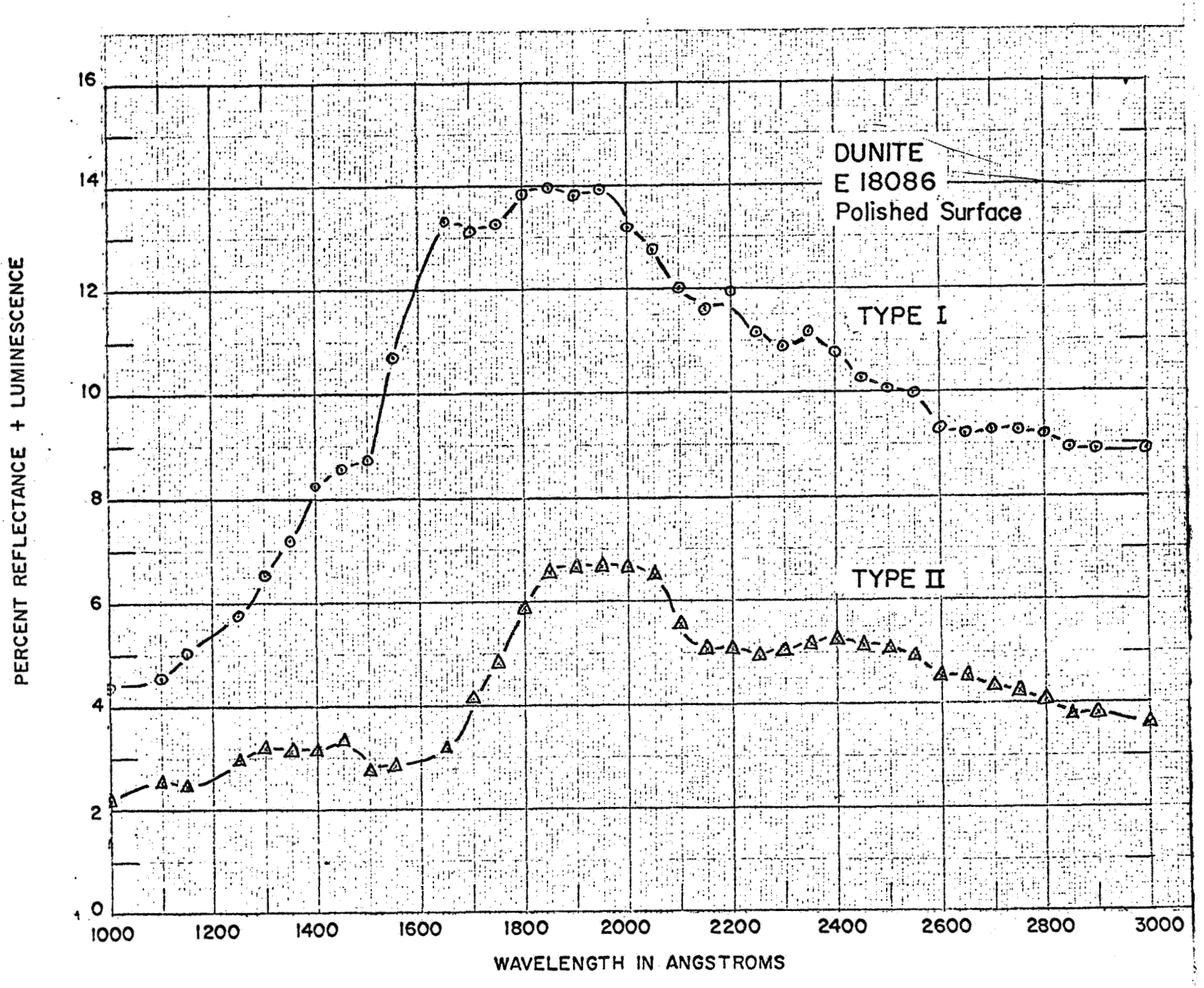
DUNITE
E 18086
Polished Surface
TYPE I
TYPE II
PERCENT REFLECTANCE + LUMINESCENCE
WAVELENGTH IN ANGSTROMS
0
2
4
6
8
10
12
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1000
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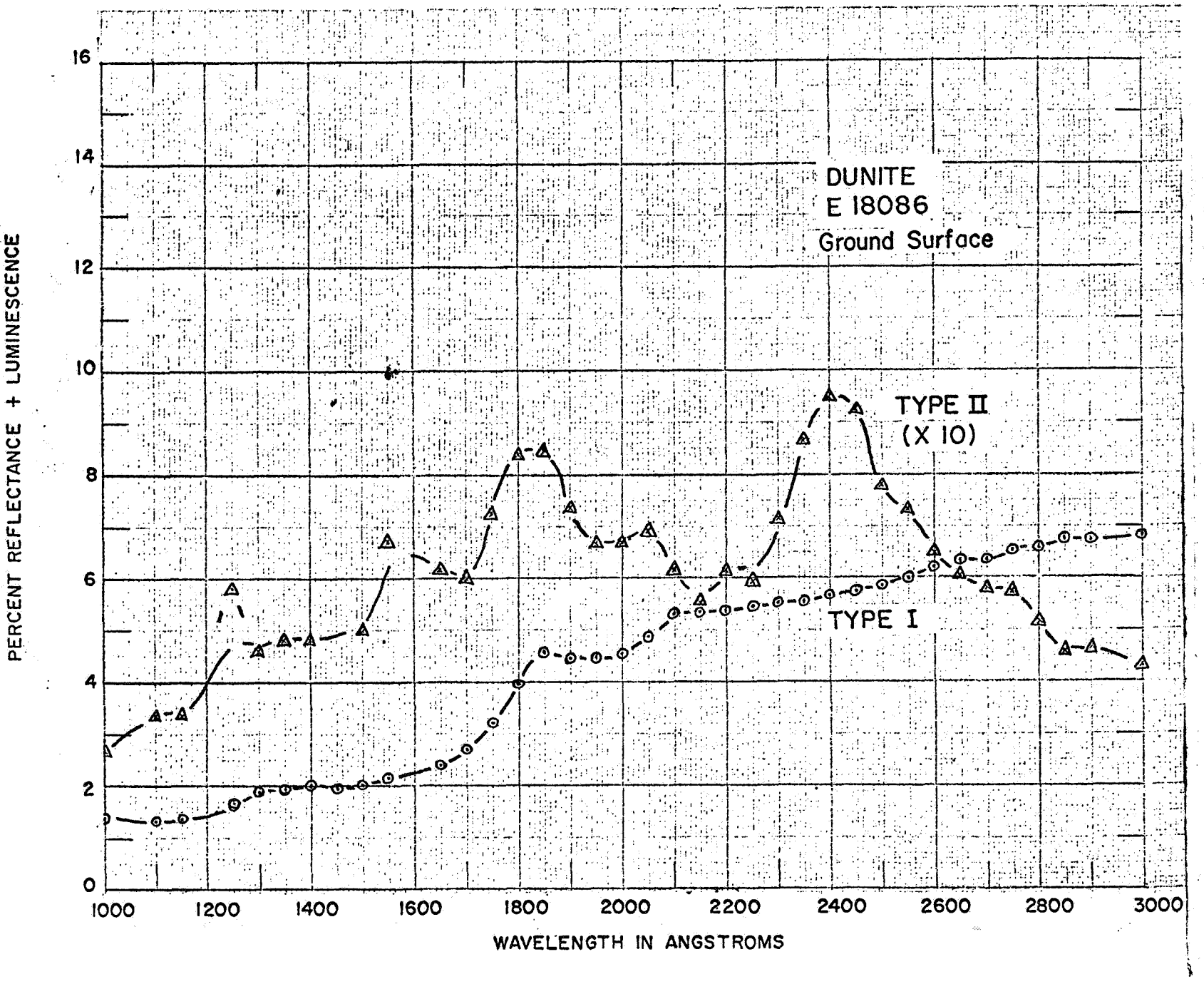
DUNITE
E 18086
Ground Surface
TYPE II
(X 10)
TYPE I
PERCENT REFLECTANCE + LUMINESCENCE
WAVELENGTH IN ANGSTROMS
0
2
4
6
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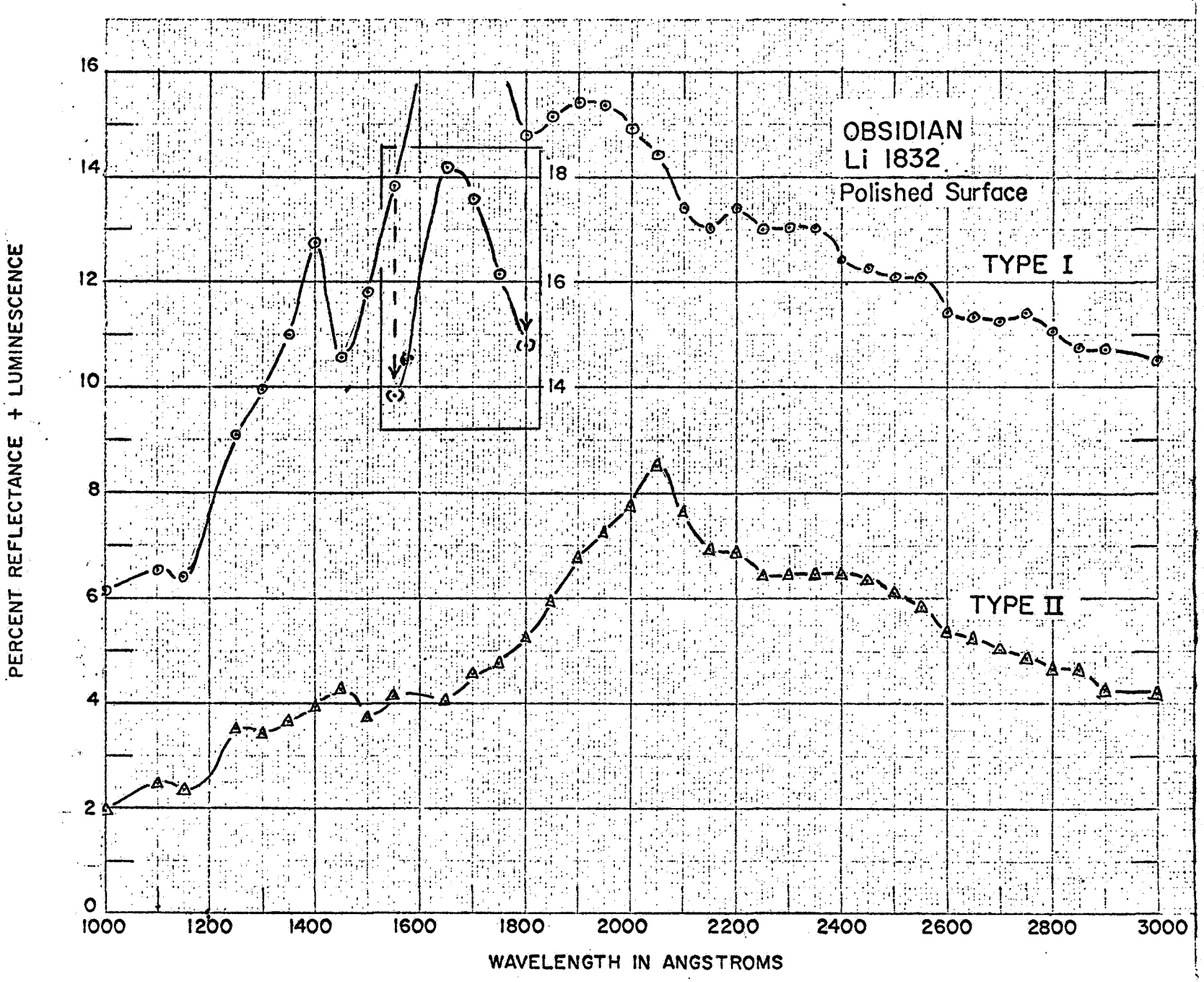
OBSIDIAN
Li 1832
Polished Surface
TYPE I
TYPE II
PERCENT REFLECTANCE + LUMINESCENCE
WAVELENGTH IN ANGSTROMS
0
2
4
6
8
10
12
14
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18
1000
1200
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2600
2800
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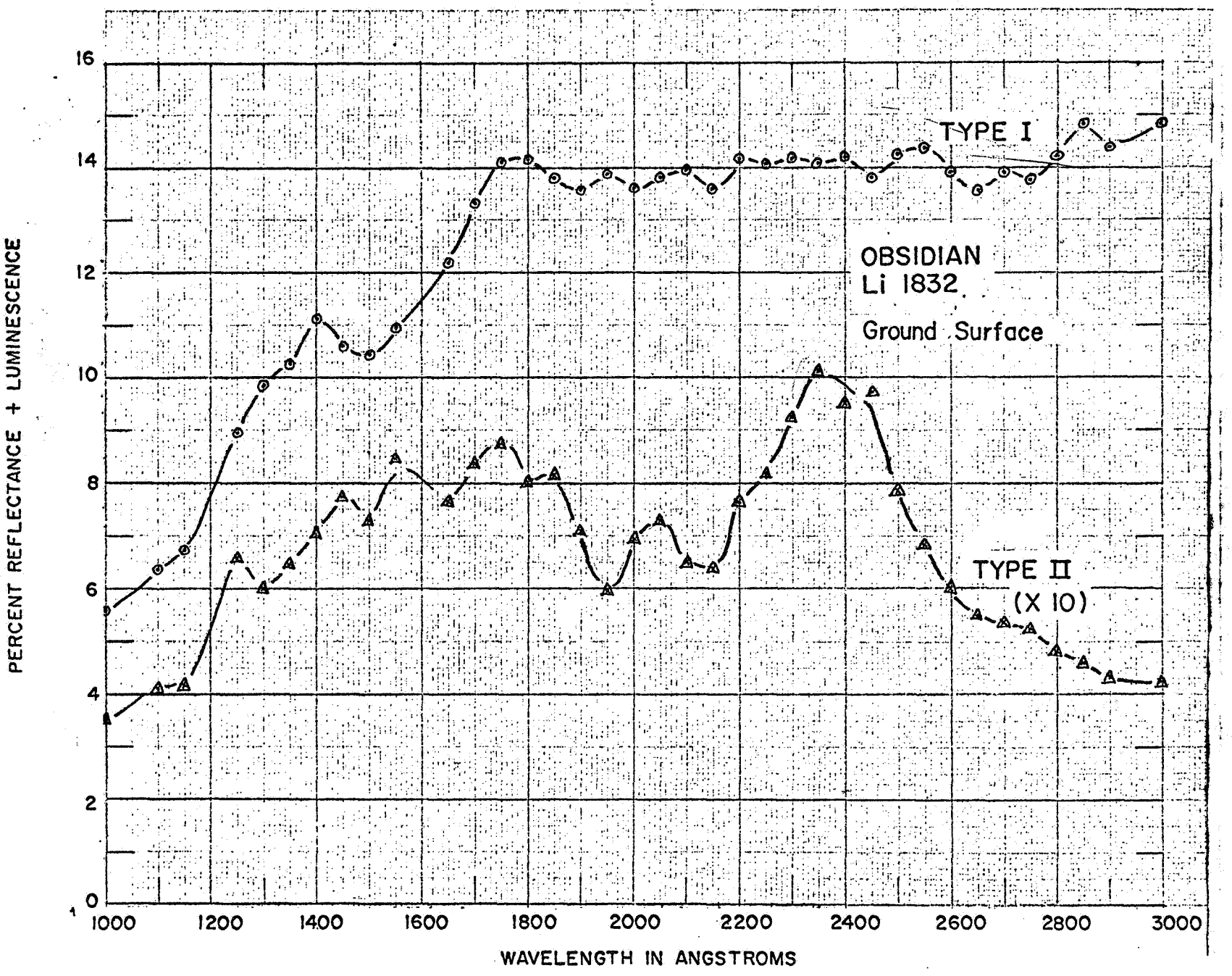
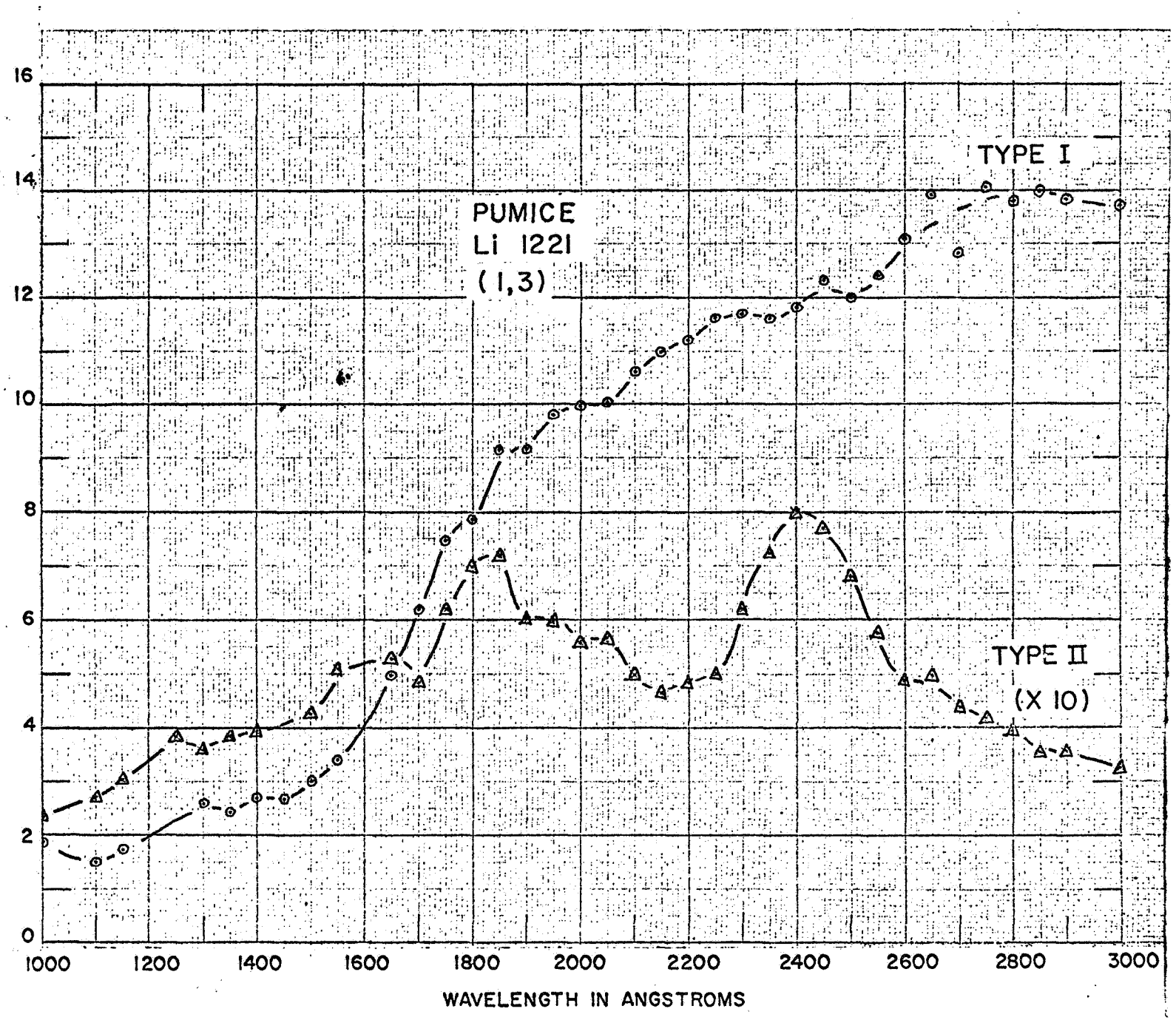

PERCENT REFLECTANCE + LUMINESCENCE
16
14
12
10
8
6
4
2
0
1000
1200
1400
1600
1800
2000
2200
2400
2600
2800
3000
WAVELENGTH IN ANGSTROMS
TYPE I
OBSIDIAN
Li 1832
Ground Surface
TYPE II
(X 10)


PUMICE
Li 1221
(1,3)
TYPE I
TYPE II
(X 10)
PERCENT REFLECTANCE + LUMINESCENCE
WAVELENGTH IN ANGSTROMS
0
2
4
6
8
10
12
14
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1000
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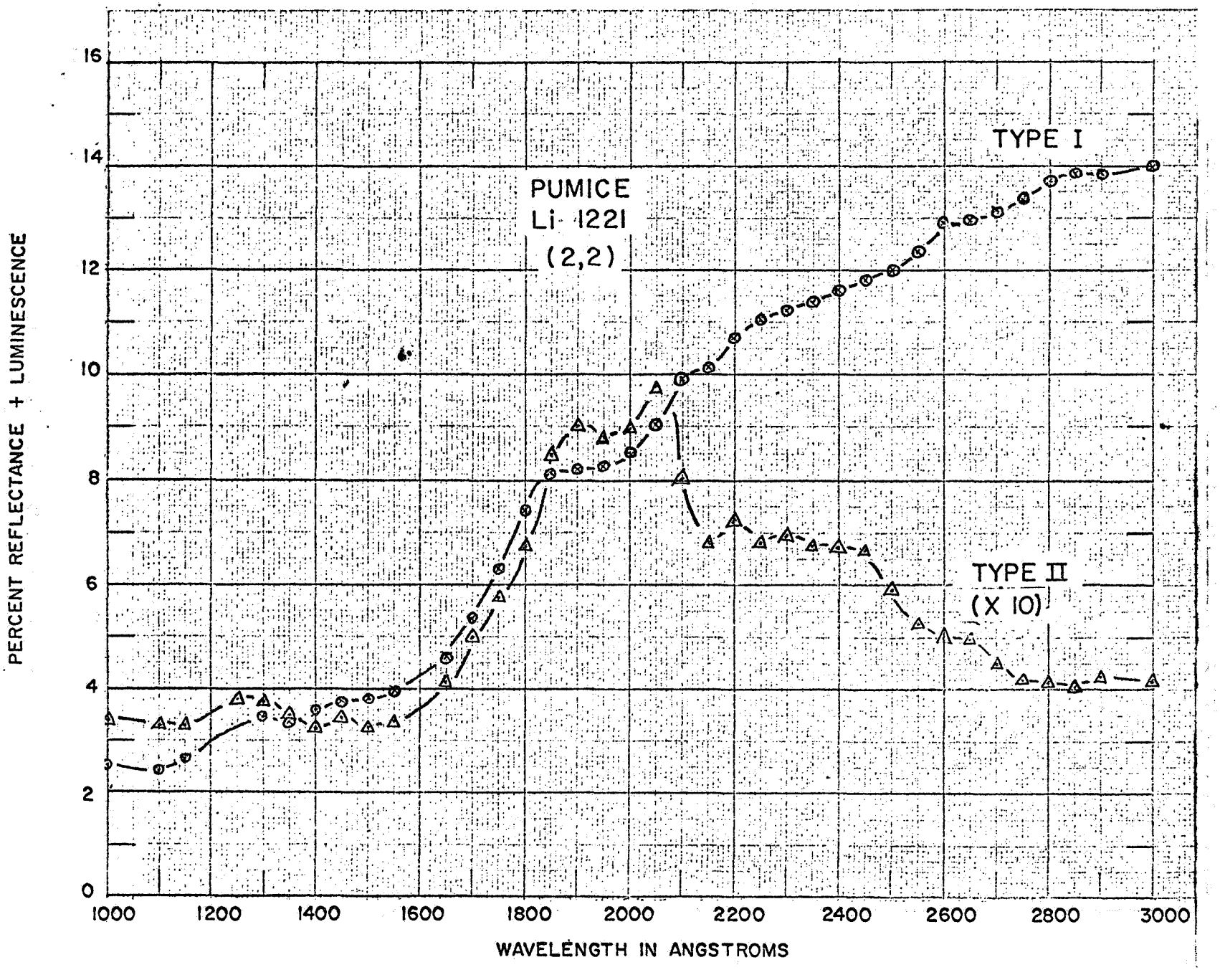
3000


PUMICE
Li-1221
(2,2)
TYPE I
TYPE II
(X 10)
PERCENT REFLECTANCE + LUMINESCENCE
WAVELENGTH IN ANGSTROMS
0
2
4
6
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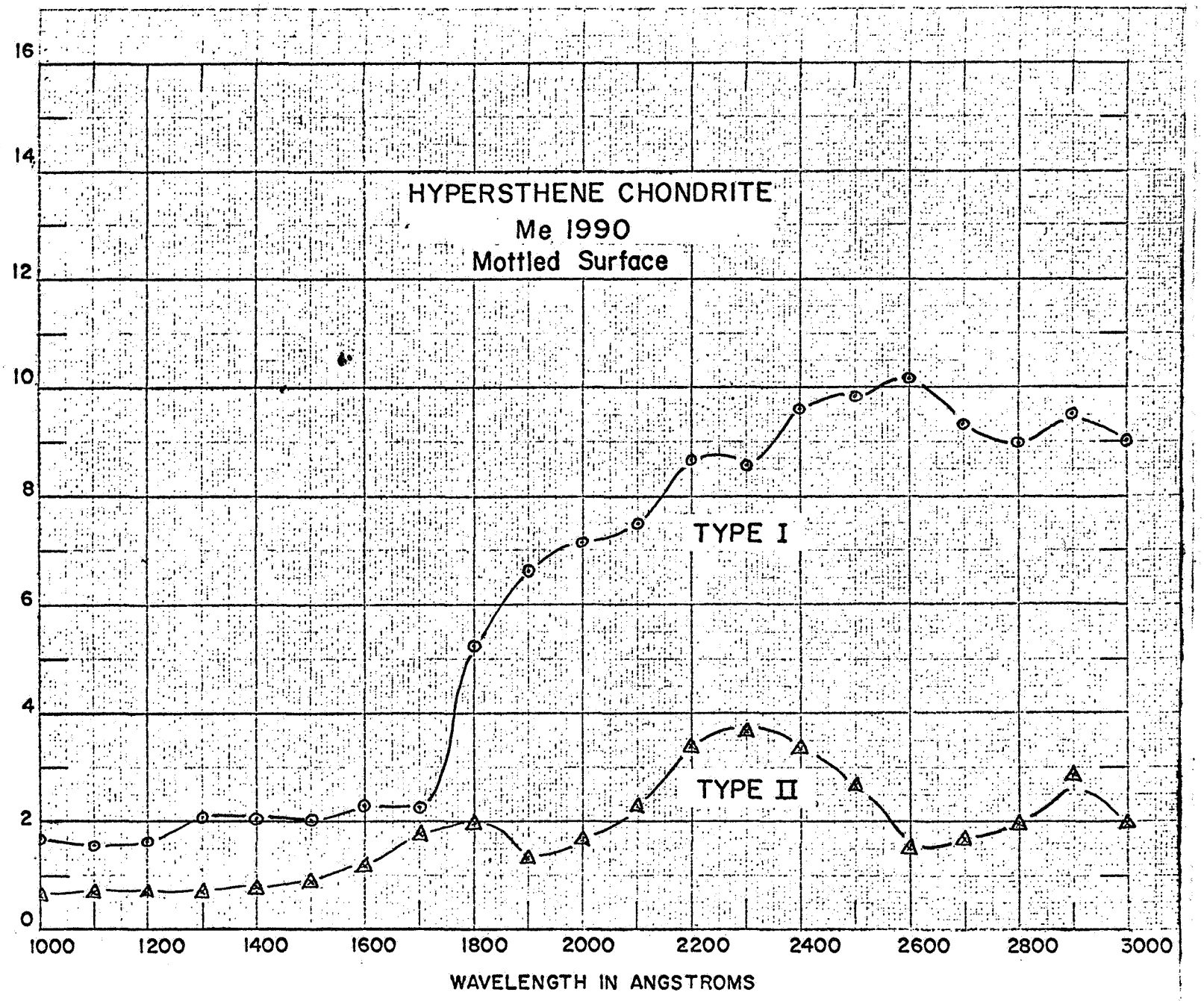
HYPERSTHENE CHONDRITE
Me 1990
Mottled Surface
TYPE I
TYPE II
PERCENT REFLECTANCE + LUMINESCENCE
WAVELENGTH IN ANGSTROMS
0
2
4
6
8
10
12
14
16
1000
1200
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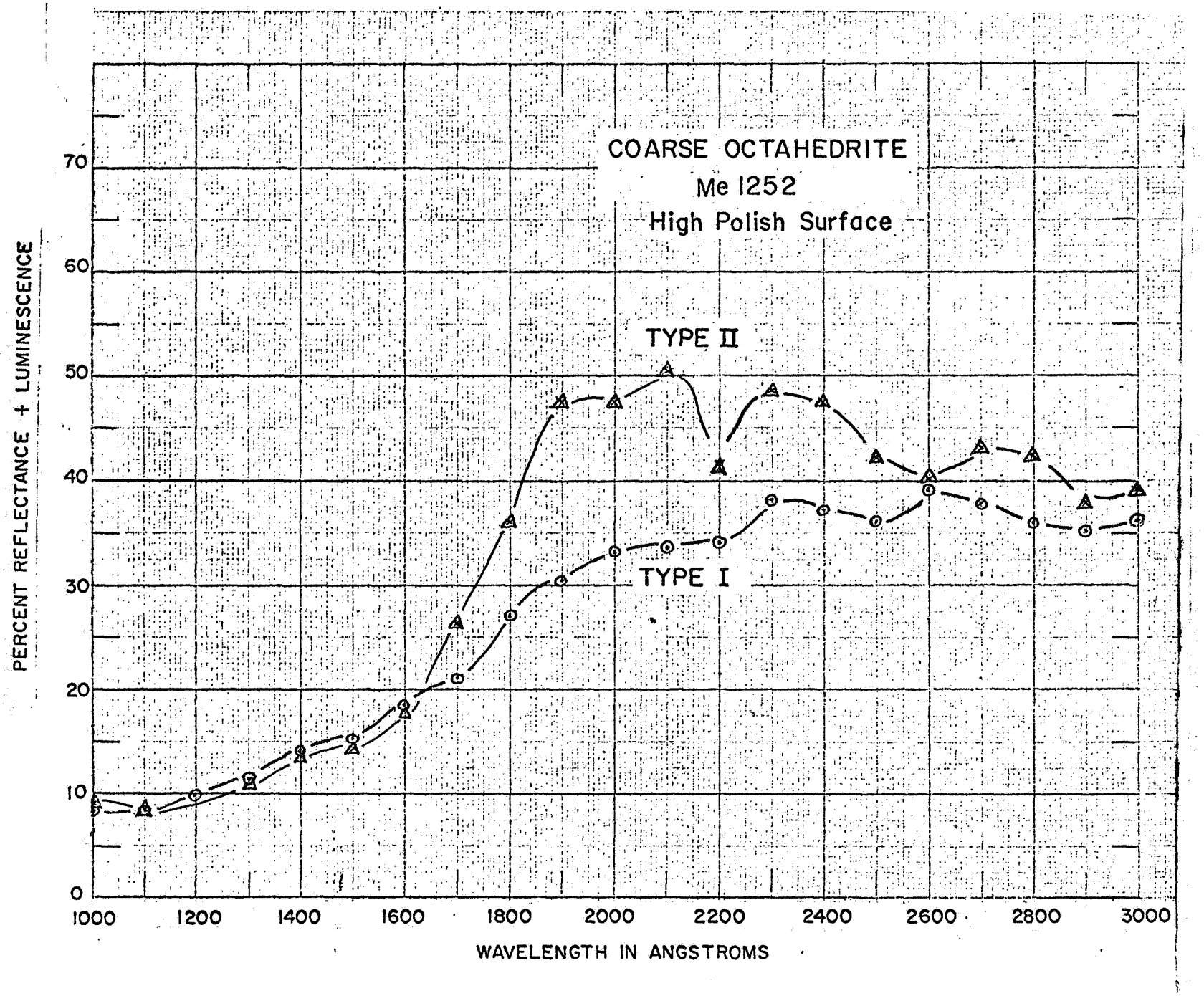
COARSE OCTAHEDRITE
Me 1252
High Polish Surface
TYPE II
TYPE I
PERCENT REFLECTANCE + LUMINESCENCE
WAVELENGTH IN ANGSTROMS
0
10
20
30
40
50
60
70
1000
1200
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3000

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